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(54) Title: ALKYLSILOXANE COMPOSITIONS

(57) Abstract

Compositions containing alkylsiloxane and at least one compound selected from the group consisting of 1,1,1,2,3,4,4,5,5,5decafluoropentane, nonafluoromethoxybutane, nonafluoroethoxybutane, dichloropentafluoropropane, 2,2-dichloro-1,1,1-trifluoroethane and isopropanol are described. These compositions are useful as cleaning agents, heat transfer media, and carrier fluids.

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ALKYLSILOXANE COMPOSITIONS

CROSS-REFERENCE TO RELATED APPLICATION

This application claims the benefit of U.S. Provisional Application No. 60/024,799 filed August 13, 1996.

FIELD OF THE INVENTION

This invention relates to compositions containing alkylsiloxanes. These compositions include alkylsiloxane and at least one compound selected from the group consisting of 1,1,1,2,3,4,4,5,5,5-decafluoropentane, nonafluoromethoxybutane, nonafluoroethoxybutane, dichloropentafluoropropane, 2,2-dichloro-1,1,1-trifluoroethane and isopropanol. These compositions are useful as cleaning agents, heat transfer media, and carrier fluids.

20 <u>BACKGROUND OF THE INVENTION</u>

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Fluorinated hydrocarbons have many uses such as cleaning agents, drying agents or carrier fluids. Such compounds include 1,1,2-trichloro-1,2,2-trifluoroethane (CFC-113). In recent years it has been pointed out that certain kinds of fluorinated hydrocarbons released into the atmosphere may adversely affect the stratospheric ozone layer. Although this proposition has not yet been completely established, there is a movement toward the control of the use and production of certain chlorofluorocarbons (CFCs) and hydrochlorofluorocarbons (HCFCs) under an international agreement. There is a parallel concern about the contribution perfluorinated compounds make to global warming, therefore, it is desirable to find compounds that are partially fluorinated to reduce potential for global warming.

Accordingly there is a demand for the development of new compounds that have lower ozone depletion potential and lower global warming contribution than existing compounds while still achieving acceptable cleaning performance.

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It is desirable to find partially fluorinated compounds for use as a cleaning agent to clean for example, silicone off medical instruments and tubing. Partially fluorinated compounds may also be useful as a carrier fluid, for example, to place a thin layer of silicone or another compound on a surface. To clean a surface containing silicone or to place silicone on a surface, it is desirable to add compounds to the partially fluorinated solvent to enhance solubility of silicone. It is particularly desirable that these compounds be non-flammable. These enhanced solvents may also be useful as heat transfer fluids, particularly in secondary loop systems. Enhanced solvents can also act as carriers for other compounds such as adhesion promoter accelerators, initiators and catalysts.

Accordingly, it has been found that adding specific agents to partially fluorinated compounds can enhance solubility of residues, for example, silicone, enhance cleaning performance, act as carrier fluids and heat transfer fluids.

SUMMARY OF THE INVENTION

The present invention relates to the following compositions: a first component, alkylsiloxane of the formula

where R is an alkyl group having from 1 to 10 carbon atoms and t is an integer from 1 to 5, and a second component, wherein the second component is selected from the group consisting of 1,1,1,2,3,4,4,5,5,5-decafluoropentane, nonafluoromethoxybutane, nonafluoroethoxybutane and dichloropentafluoropropane and optionally, a third component, wherein the third component is selected from the group consisting of isopropanol and 2,2-dichloro-1,1,1-trifluoroethane.

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These compositions may be useful as cleaning agents, heat transfer media, silicone removal agents, and as carrier fluids for silicone, adhesion promoters or other compounds.

DETAILED DESCRIPTION

The present invention relates to the discovery of mixtures of alkylsiloxane and at least one of 1,1,1,2,3,4,4,5,5,5-decafluoropentane, nonafluoromethoxybutane, nonafluoroethoxybutane, dichloropentafluoropropane, isopropanol and 2,2-dichloro-1,1,1-trifluoroethane.

1-99% of each of the above components can be used as cleaning agents, heat transfer media, silicone removal agents, and as carrier fluids for compounds such as silicone, adhesion promoters or other compounds.

Nonafluoromethoxybutane (C₄F₉OCH₃) isomers of the present invention include 1,1,1,2,2,3,3,4,4,-nonafluoro-4-methoxy-butane (CH₃OCF₂CF₂CF₂CF₃), 1,1,1,2,3,3,-hexafluoro-2-(trifluoromethyl)-3-methoxy-propane (CH₃OCF₂CF(CF₃)₂),

20 1,1,1,3,3,3-hexafluoro-2-methoxy-2-(trifluoromethyl)-propane (CH₃OC(CF₃)₃), and 1,1,1,2,3,3,4,4,4-nonafluoro-2-methoxy-butane (CH₃OCF(CF₃)CF₂CF₃), approximate isomer boiling point = 60°C;

Nonafluoroethoxybutane ($C_4F_9OC_2H_3$) isomers of the present invention include 1,1,1,2,2,3,3,4,4-nonafluoro-4-ethoxybutane ($CH_3CH_2OCF_2CF_2CF_2CF_3$),

1,1,1,2,3,3-hexafluoro-2-(trifluoromethyl)-3-ethoxypropane (CH₃CH₂OCF₂CF(CF₃)₂), l,1,1,3,3,3-hexafluoro-2-ethoxy-2-(trifluoromethyl)-propane (CH₃CH₂OC(CF₃)₃), and l,1,1,2,3,3,4,4,4-nonafluoro-2-ethoxybutane (CH₃CH₂OCF(CF₃)CF₂CF₃) with approximate isomer boiling points of 73°C;

Dichloropentafluoropropane isomers include 1,1-dichloro-2,2,3,3,3-30 pentafluoropropane (CHCl₂CF₂CF₃, HCFC-225ca), boiling point = 50°C, and 1,3-dichloro-1,1,2,2,3-pentafluoropropane (CHClFCF₂CClF₂, HCFC-225cb), boiling point = 56°C. 2,2-dichloro-1,1,1-trifluoroethane (HCFC-123 or CHCl₂CF₃) has a boiling point of 27°C. Isopropanol (CH₃)₂CH0H has a boiling point of 82.2°C.

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Effective amounts of these compositions can contain from 10 to 60 weight percent alkylsiloxane and 40 to 90 weight percent of at least one of HFC-43-10mee, C₄F₉OCH₃, C₄F₉OC₂H₅, HCFC-225ca, or HCFC-225cb. Effective amounts can also contain 1-30 weight percent alkylsiloxane, 9-50 weight percent HCFC-123, and 25-90 weight percent of at least one of HFC-43-10mee, C₄F₉OCH₃, C₄F₉OC₂H₅, HCFC-225ca, or HCFC-225cb. Compositions may also contain effective amounts of 10-59 weight percent alkylsiloxane, 1-20 weight percent isopropanol, and 40-89 weight percent of at least one of HFC-43-10mee, C₄F₉OCH₃, C₄F₉OC₂H₅, HCFC-225ca, or HCFC-225cb.

Specific examples illustrating the invention are given below. Unless otherwise stated therein, all percentages are by weight. It is to be understood that these examples are merely illustrative and in no way are to be interpreted as limiting the scope of the invention.

EXAMPLE 1

Flammability Test

A Penski-Martin Closed Cup flash point tester was filled with mixtures shown in Table 1. OS-10 is hexamethylsisiloxane and OS-20 is octamethyltrisiloxane. Flash points were determined in a temperature range from about -20C to 38C. No flash points were observed at any temperature for any mixture tested. Flammability was also tested by attempting to ignite mixtures in a pan with a spark and a flame. No ignitions resulted in mixtures tested.

TABLE 1

Flammability Data

	Muxture Wt% F	lash Point Ignition in Pa	n
	HFC-43-10mee/		
	80/20	None	
10	70/30	None	
	60/40	None	
	50/50	None	
	HFC-43-10mee/	HCFC-123/OS-10	
	50/30/20	None	None
15	60/20/20		None
	55/25/20		None
	60/25/15		None
	45/35/20	None	None
	45/30/25	None	None
20	HFC-43-10mee/		
	50/40/10	• •	None
	60/30/10		None
	70/20/10		None

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EXAMPLE 2

A suitable container was filled with mixtures shown in Table 2 and mixed thoroughly at room temperature. Trans-1,2-DCE is trans-1,2-dichloroethylene (CCI=CCI). Dow Corning 360, Dow Corning 550, or Dow Corning 1107 medical grade silicone oil was then slowly added to each mixture until the blend would no longer dissolve the oil.

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TABLE 2

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	<u>Mixture</u>	Weight Percent	Wt. % S	Silicone Dis	solved
			DC-360	DC-550 D	C-1107
	CFC-113	100	16.0		
	Hexane	100	16.0		
10	43-10mee/ cyclopentane/	65/15/20	0.5		
	trans-1,2-dichloroethyler	ne			
	43-10mee/ cyclohexane/	85/5/10	0.5		
15	trans-1,2-dichloroethyler	ne			
	43-10mee/OS-10	60/40	10.02		
		50/50	23.0		
	43-10mee/123/OS-10	50/30/20	5.12	14.0	25.0
		60/25/15	<1.0	1.68	9.0
20		60/20/10	<1.0	3.01	9.0
		47.5/35/17.5	5.0	12.0	24.0
		45/30/25	18.2		
		50/35/15	5.5		
		45/35/20	12.85		
25	43-10mee/OS-20/ isopropanol	50/40/10	3.5		

Results show that the addition of hexamethyldisiloxane to HFC-43-10mee significantly improves silicone solubility. Alkysyloxane-containing blends can also act as silicone carrier fluids due to their ability to dissolve silicone. These blends also have the advantage of being non-flammable and non-toxic.

EXAMPLE 3

A suitable container was filled with the mixtures shown in Table 3. Preweighed tubing made of polyurethane was immersed in each mixture at a specified temperature for three minutes. Parts were then dried, weighed and observed for swelling.

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TABLE 3 Weight Gain and Enlargement

	Pre-Test Post-Test			% Wt Ch	ange in	
	Solvent	Wt (kg)	Wt (kg)	BP(°C)	Increase	Dimension
	CFC-113	0.1917	0.213	47	11.1	Enlarged
10	HFC-					_
	43-10mee/	0.174	0.2209	37	27.0	Enlarged and
	cyclopentane/					softened
	trans-1,2-DCE					
	(65/15/20 wt%)					
15	Hexane	0.1669	0.2112	. 68	26.5	Enlarged
	HFC-43-10mee/	0.1481	0.1481	55.5	0.0	No effect
	123/OS-10					
	(85/15 wt%)					

Results show the blends containing alkylsiloxanes demonstrated significantly reduced weight gain and swelling versus the other compositions tested.

EXAMPLE 4

Polyurethane, nylon and ABS tubing were coated with silicone and then cleaned with a solution containing 85 weight percent HFC-43-10mee and 15 weight percent hexamethyldisiloxane. Results are shown in Table 4 below.

TABLE 4

Cleanability

	Tubing Type	Degree of Cleaning
30	Polyurethane	Visually Clean
	ABS	Visually Clean
	Nylon	Visually Clean

Tubing also felt clean with no evidence of silicone remaining on the tubes.

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EXAMPLE 5

Lengths of Polysilicone tubing (Boston Scientific) were cut with tube weight and diameter recorded. A suitable container was filled with solvent compositions as shown in Table 5. Each composition was heated to the vapor temperatures shown and tubing was immersed in the solvent for several minutes. Tubing was removed and allowed to dry for 30 seconds. Tubing was then reweighed and the diameter remeasured. The change in weight and diameter were calculated.

TABLE 5

			Change in	Change in	Vapor
15		Wt%	Weight (g)	Diameter(in)	T(°C)
	3 minute immersion:				
	HCFC-225	100	+0.123	+0.016	53.5
	CFC-113	100	+0.1738	+0.024	47.5
	HFC-43-10mee/OS-10	50/50	+0.0778	+0.023	57.0
20	HFC-43-10mee/OS-20	50/50	+0.0942	+0.031	57.0
	HCFC-225	100	+0.1182	+0.016	21.0
	CFC-113	100	+0.0874	+0.020	**
	HFC-43-10mee/OS-10	50/50	+0.0713	+0.018	10
	HFC-43-10mee/OS-20	50/50	+0.0611	+0.023	"
25	15 minute immersion:				
	CFC-113	100	+0.3710	+0.038	21.0
	HFC-43-10/123/OS-10	50/30/20	+0.1595	+0.026	11
	HFC-43-10/OS-20/IPA	50/40/10	+0.1235	+0.023	Ħ
	HFC-43-10/OS-20/IPA	60/20/10	+0.1031	+0.021	н
30	HFC-43-10/OS-20/IPA	70/20/10	+0.0871	+0.019	ti

For connecting polysilicone tubing using a solvent, it is desirable to have a significant increase in tube diameter without a large increase in tube weight. The samples containing alkysiloxanes generally showed adequate change in tube diameter and the smallest increase in tube weight.

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ADDITIONAL COMPOUNDS

Other components, such as aliphatic hydrocarbons having a boiling point of 0-100°C, hydrofluorocarbonalkanes having a boiling point of 0-100°C, hydrocarbon esters

10 having a boiling point between 0-100°C, hydrochlorofluorocarbons having a boiling point between 0-100°C, hydrochlorofluorocarbons having a boiling point of 0-100°C, hydrochlorocarbons having a boiling point of 0-100°C, hydrochlorocarbons having a boiling point between 0-100°C, chlorocarbons and perfluorinated compounds, can be added to the azeotropic or azeotrope-like compositions described above without substantially changing the properties thereof, including the

15 constant boiling behavior, of the compositions. Examples of such components, which typically do not exceed about 10 weight percent of the total composition, include the following:

	COMPOUND	FORMULA	boiling point, °C
20	HCFC-123	CHCl2CF3	27
	HCFC-141b	CFCl ₂ CH ₃	32
	HCFC-225aa	CHF2CCl2CF3	53
	HCFC-225da	CClF2CHClCF3	. 50
	HFC-HFC-43-10mf	CF ₃ CH ₂ CF ₂ CF ₂ CF ₃	52
25	HFC-HFC-43-10mcf	CF ₃ CF ₂ CH ₂ CF ₂ CF ₃	52
	FC-C-51-12	$cyclo-C_4F_6(CF_3)_2$	45
		CH,OCF2CHFCF,	52
	HFC-C-354	cyclo-CF2CF2CH2CH2	50
		C ₄ F ₉ CH=CH ₂	58
30	MEK	CH ₃ C(O)C ₂ H ₅	80
	THF	cyclo-OC ₄ H ₈	66
	methyl formate	HC(O)OCH ₃	32
	ethyl formate	HC(O)OC ₂ H ₅	54
	methyl acetate	CH ₃ C(O)OCH ₃	56
35	ethyl acetate	CH ₃ C(O)OC ₂ H ₅	77
	1,2-dichloroethane		84
	acetonitrile		82
	methylene chloride		40
	heptane	$CH_3(CH_2)_5CH_3$	98
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Additives such as lubricants, corrosion inhibitors, stabilizers, surfactants. dyes and other appropriate materials may be added to the novel compositions of the invention for a variety of purposes provided they do not have an adverse influence on the composition, for their intended applications. Examples of stabilizers include nitromethane and nitroethane.

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CLAIMS

What is claimed is:

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- 1. A composition comprising effective amounts of alkylsiloxane and at least one compound selected from the group consisting of 1,1,1,2,3,4,4,5,5,5-decafluoropentane, nonafluoromethoxybutane, nonafluoroethoxybutane, dichloropentafluoropropane, 2,2-dichloro-1,1,1-trifluoroethane and isopropanol.
- The composition of claim 1 comprising 10-60 weight percent
 alkylsiloxane and 40-90 weight percent 1,1,1,2,3,4,4,5,5,5-decafluoropentane; 10-60 weight percent alkylsiloxane and 40-90 weight percent nonafluoroethoxybutane; 10-60 weight percent alkylsiloxane and 40-90 weight percent 1,1-dichloro-2,2,3,3,3-pentafluoropropane; 10-60 weight percent alkylsiloxane and 40-90 weight percent 1,3-dichloro-1,1,2,2,3-pentafluoropropane; 10-60 weight percent alkylsiloxane, 1-89 weight percent 1,1-dichloro-2,2,3,3,3-pentafluoropropane and 1-89 weight percent 1,3-dichloro-1,1,2,2,3-pentafluoropropane.
- 3. The composition of Claim 1 comprising 1-30 weight percent
 alkylsiloxane, 9-50 weight percent 2,2-dichloro-1,1,1-trifluoroethane, and 25-90 weight
 percent of at least one compound selected from the group consisting of
 1,1,2,3,4,4,5,5,5-decafluoropentane, nonafluoromethoxybutane,
 nonafluoroethoxybutane, 1,1-dichloro-2,2,3,3,3-pentafluoropropane and 1,3-dichloro1,1,2,2,3-pentafluoropropane.
 - 4. The composition of Claim 1 comprising 10-59 weight percent alkylsiloxane, 1-20 weight percent isopropanol, and 40-89 weight percent of at least one compound selected from the group consisting of 1,1,1,2,3,4,4,5,5,5-decafluoropentane,

- 5 nonafluoromethoxybutane, nonafluoroethoxybutane, 1,1-dichloro-2,2,3,3,3-pentafluoropropane and 1,3-dichloro-1,1,2,2,3-pentafluoropropane.
- 5. A composition comprising 1-30 weight percent hexamethyldisiloxane,
 25-90 weight percent 1,1,1,2,3,4,4,5,5,5-decafluoropentane and 9-50 weight percent 2,2dichloro-1,1,1-trifluoroethane; and 10-59 weight percent octamethyltrisiloxane, 40-89
 weight percent 1,1,1,2,3,4,4,5,5,5-decafluoropentane and 1-20 weight percent
 isopropanol.
- 6. A process for cleaning a solid surface which comprises cleaning said surface with a composition of claim 1 or 5.
 - 7. A process for carrying an active ingredient in a solvent in which the solvent comprises a composition of claim 1 or 5.
 - 8. A process for transferring heat from a heat source to a heat sink using a composition of claim 1 or 5.
 - 9. A fluid for carrying silicone comprising a composition of claim 1 or 5.

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INTERNATIONAL SEARCH REPORT

Inte Nonel Application No PC 1/US 97/14213

A. CLASSIFICATION OF SUBJECT MATTER IPC 6 C11D7/50 C11D7/26 C11D7/28 According to International Patent Classification (IPC) or to both national classification and IPC B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) IPC 6 C11D Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Electronic data base consulted during the international search (name of data base and, where practical, search terms used) C. DOCUMENTS CONSIDERED TO BE RELEVANT Category * Citation of document, with indication, where appropriate, of the relevant passages Relevant to claim No. EP 0 699 746 A (KABUSHIKI KAISHA TOSHIBA) Х 1,2,6,7 6 March 1996 see page 24; example 29; table 10 see page 25; example 30; table 11 see page 11, line 50 - page 12, line 2 EP 0 576 687 A (OLYMPUS OPTICAL CO. LTD.) X 1,6,7 5 January 1994 see page 10 - page 11; examples 7,8; table EP 0 710 715 A (AG TECHNOLOGY CO. LTD.) 8 Α 1 May 1996 see page 1, line 1 - line 38 see page 3, line 50 - line 51 see page 4, line 26 - line 34 Further documents are listed in the continuation of box C. Patent family members are listed in annex. * Special categories of cited documents : TT later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier document but published on or after the international "X" document of particular relevance; the claimed invention filing date cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "L" document which may throw doubts on priority claim(a) or which is cited to establish the publication data of another citation or other special reason (as apecified) "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such docu-*O* document referring to an oral disclosure, use, exhibition or ments, such combination being obvious to a person skilled in the art. *P* document published prior to the international filing date but later than the priority date claimed "&" document member of the same patent family Date of the actual completion of the international search Date of mailing of the international search report 2 4. **11**. 97 11 November 1997 Name and mailing address of the ISA Authorized officer European Patent Office, P.B. 5818 Patentiaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Ketterer, M Fax: (+31-70) 340-3016

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Inte onet Application No
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